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Mechanical and physical characterization of sodium hydroxide treated Borassus fruit fibers

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Abstract: In order to improve the properties by chemical modification and to optimize the alkali concentration, we treated Borassus fruit fine fibers with aqueous sodium hydroxide solutions of different concentrations. In each case, the tensile properties of the fibers were determined. The morphology of the untreated and alkali treated fibers was observed using scanning electron microscope. The surface of the fibers became rough on alkali treatment. The tensile properties of the fibers improved on alkali treatment. The fibers attained maximum tensile properties when treated with 15% aqueous sodium hydroxide solution and decreased thereafter. The crystallinity index of the fibers showed a similar trend. The thermal stability of the alkali treated fibers was found to be higher than that of untreated fibers. Further, the char content was maximum for fibers treated with alkali having concentration of 15% and above. The chemical composition indicated that the percentageof α-cellulose was maximum when the fibers were treated with 15% aqueous sodium hydroxide solution and then decreased thereafter thus indicating the beginning of degradation of the fibers at higher concentrations. Thus, the optimum concentration of NaOH was established as 15% for alkali treat-

Keywords: borassus fruit fine fiber; chemical composition; mercerization; spectral analysis; tensile properties; thermal properties.

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Introduction

Due to the global environment awareness, depletion of oil resources and stringent environmental regulations, a need is felt to develop environment friendly Green composites (Li et al. 2004; John and Thomas 2008). Plant fibers are spread worldwide, and form a source of renewable materials that are mainly abundant in the tropical areas. Further, with plant fibers, biodegradability can contribute to a healthier ecosystem and their low cost and reasonable performance fulfill economic interest of various industries (Pickering et al. 2007; Reddy and Yang 2005). Natural fibers obtained from the various parts of the plants are known as vegetable fibers. These fibers are classified into five categories depending on the part of the plant from which they are extracted. A. Bast or Stem fibers - jute (Gassan and Bledzki 1999), hibiscus (Singha and Thakur 2008), hemp (Pickering et al. 2007) etc., B. Leaf fibers - sisal (Joseph et al. 1996), pineapple (George et al. 1998), coconut leaf sheath (Reddy et al. 2010) etc., C. Seed/Fruit fibers - cotton (Bledzki and Gassan. 1999), coir (Rout et al. 2001), tamarind (Maheswari et al. 2008) etc., D. Grass fibers bamboo (He et al. 2007) napier (Reddy et al. 2009a) etc., and E. Bark fabrics - hildegardia (Rajulu et al. 2002), streculia (Ramudu et al. 2009) etc. Plant fibers are mainly composed of cellulose, hemi cellulose, and lignin. Other components, usually regarded as surface impurities, are pectin and wax substances (John and Anandjiwala. 2008). The α-cellulose structurally consists of Dglucopyranose units linked by β -position whereas hemicellulose consists of similar units of α -cellulose with branching (Pandey. 1999). As a result of branching; hemicellulose exists in an amorphous form while α-cellulose is crystalline. On the other hand, lignin is a polymer consisting of phenolic units with alkoxy functional groups. In recent years, the use of natural fibers as reinforcement or filler in the thermoplastic and thermosetting matrices is of great interest to many researchers (Li et al. 2004; John and Thomas 2008; Pickering et al. 2007; George et al. 1998; Ghasemi et al. 2008). These fibers have many advantages, such as low density, high specific strength and modulus (John and Anandjiwala 2008), relative non-abrasiveness, ease of fiber sur-



face modification, and wide availability (Bolton 1995). Natural fibers are much cheaper than synthetic fibers and can replace synthetics in many applications where cost outweighs high composite performance requirements.

The main disadvantages of natural fibers in composites are the allowable lower processing temperatures, incompatibility between hydrophilic natural fibers and hydrophobic polymers and potential moisture absorption. Further, lack of good interfacial adhesion and poor resistance to moisture absorption makes the use of natural fiber-reinforced composites less attractive. Various fiber surface treatments like mercerization, isocyanate treatment, acrylation, permanganate treatment, acetylation, silane treatment and peroxide treatment have been carried out which may result in improving composite properties (Gassan and Bledzki 1999; Singha and Thakur 2008; Rout et al. 2001; Ray and Sarkar 2001; Mwaikambo and Ansell 2002; Bledzki et al. 1996). Research on a cost effective modification of natural fibers is necessary since the main attraction for today's market of biocomposites is the competitive cost of natural fibers. Interfaces play an important role in the physical and mechanical properties of composites. Surface treatment of reinforcing fibers is normally carried out to improve their compatibility with the polymer matrix. Several researchers have also reported the improvement of the mechanical properties of cellulose fibers when alkalized for different times and at different alkali concentrations (Ray and Sarkar 2001; Rout et al. 2001). Most of the impurities existing on the fiber surfaces and part of the amorphous cellulose component can be removed by alkali treatment. The alkali treatment changes the chemical composition and modifies the crystalline structure of natural fibers (Bledzki et al. 1996).

The authors (Reddy et al. 2009b) already studied the basic properties of the Borassus fibers and found them suitable as natural reinforcement for biocomposites. In the present work, an attempt was made to investigate the effect of alkalization with different concentrations on the properties of Borassus fruit fine fibers on their properties. The changes occurring in borassus fine fibers after treatment with 2%, 5%, 10%, 15% and 20% NaOH solution for 30 min were investigated. The fibers were characterized using chemical analysis, FT-IR, XRD and TGA techniques. The surface changes were examined using scanning electron microscope. Tensile testing was used to characterize the mechanical properties of the fibers. At present, the Borassus fibers have limited applications in view of the moderate mechanical properties. The authors carried out the present work to improve the properties of the Borassus fruit fibers by chemical methods so that they may find applications in the manufacturing of Green composites.

Materials and methods

Materials

Fine fibers were extracted from Borassus fruits and commercial grade sodium hydroxide pellets (Merck, India), benzene, sodium chlorite, acetic acid, sodium bisulphate, and ethanol (SD Fine



Extraction of the fibers from the fruits

The Borassus fruit belongs to Arecaceae family with *Borassus flabellifer L* species. The Borassus fibers were extracted from dried ripened fruits. The fruits were dipped in water for two weeks and the black skin was separated. Below it, two types of fibers - long coarse and short fine fibers were found. Coarse fibers were present in the fruit nut edge to edge, whereas the fine fibers were adhered to the shell of the fruit. Both types of fibers were thoroughly washed with tap water followed by distilled water and dried in the sun for a week. Then the fibers were kept in hot air oven for 24 h at $105-110^{\circ}$ C to remove the moisture. In the present work, the Borassus fine fibers were selected, as they possessed better tensile properties (Reddy et al. 2009b).

Alkali treatment

Borassus fine fibers were treated with different concentrations of aqueous sodium hydroxide solutions (2%, 5%, 10%, 15% and 20% weight/volume) at 30°C, maintaining a liquor ratio of 30:1, for 30 min to remove the hemicellulose and other greasy materials. Then the fibers were washed with water repeatedly, neutralized with dilute acetic acid, and again washed with distilled water before drying in hot air oven for a period of 24 h.

FT-IR spectral analysis

The untreated and alkali treated fibers were cryogenically cooled and powdered separately. These powders were diluted to 1% using potassium bromide (KBr) and pellets were prepared. The FT-IR spectra of the untreated and alkali treated samples were recorded in the 4000-500 cm⁻¹ region on a Perkin Elmer 16PC FT-IR instrument with 32 scans in each case at a resolution of 4 cm⁻¹.

Chemical analysis

The chemical analysis of the fibers was carried out as per the standard procedure (Chattopadhyay and Sarkar 1946; Sarkar et al. 1948) briefly described next. The preweighed chopped fibers were dewaxed with a mixture of benzene/ethanol taken in the ratio of 2:1 at 70°C for 3 h. The dewaxed fibers were washed with ethanol for 30 min, dried and weighed. The dewaxed preweighed samples were boiled for two hours in 0.7% ag. sodium chlorite solution (adjusted to a pH 4 using buffer solution) by maintaining a fiber to liquor ratio of 1:50. Later they were washed with 5% aq. sodium bisulphate solution and distilled water and dried at 105°C in a hot air oven. In this step, the lignin was removed and the weight difference after this step corresponds to the lignin content. The remaining holocellulose (hemicellulose and α-cellulose) was treated with 17.5% aqueous sodium hydroxide solution to eliminate alkali soluble hemicellulose. The weight loss and remaining weight corresponded to the contents of hemicellulose and α-cellulose respectively.



X-ray analysis

The wide-angle X-ray diffraction spectra of the fibers were recorded on a Rigaku Dmax 2500 diffractometer (Tokyo, Japan). The system has a rotating anode generator with a copper target and wide-angle powder goniometer. The generator was operated at 40 KV and 150 mA. All the experiments were performed in the reflection mode at a scan speed of 4° /min in steps of 0.05° . All samples were scanned in 2θ range varying from 5 to 50° .

Morphology

The scanning electron micrographs of the surface of the fibers were recorded on a JEOL JSM 820 microscope (Akishima, Japan). The fiber samples were gold coated before recording the micrographs.

Thermo gravimetric analysis

The thermograms of the fibers were recorded on a Perkin Elmer TGA-7 instrument in nitrogen atmosphere at a heating rate of 10°C/min in the temperature range of 30–500°C.

Tensile properties

The tensile properties, such as maximum stress, Young's modulus, and % elongation at break of the fibers, were determined using an INSTRON 3369 Universal Testing Machine (Norwood, MA) at a crosshead speed of 10 mm/min maintaining a gauge length of 50 mm. In each case, 10 samples were used and the average values is reported.

Results and Discussion

When lignocellulose fibers are treated with alkali, a major modification occurs in the form of break of hydrogen bonding in the network structure as shown in the following reaction (Mwaikambo and Ansell. 2002).

Fiber-OH + NaOH
$$\longrightarrow$$
 Fiber -O Na + H₂O + Surface impurities (1)

Alkali treatment results in a higher amount of swelling, leading to the formation of new Na-cellulose lattice, and the conversion of cellulose OH-groups to ONa-groups, expanding the molecular dimensions. Subsequent rinsing with water removes the linked Na-ions and converts into higher crystalline cellulose (Weyenberg et al. 2006).

Scanning electron micrographs of untreated and different concentrated alkali treated fibers are shown in Fig. 1. From the Figure 1 it can be observed that the cellular structures on the surface of the fibers ran longitudinally with more or less parallel orientations. The intercellular gaps, in the form of shallow longitudinal cavities, can be clearly noticed as the unit cells were partially exposed in alkali treated fibers. The intercellular cells were

bound together by cellulose, hemicellulose and lignin. The micrographs of alkali treated fibers show a large number of regularly placed holes or pits on the surface. These are most possibly from the removal of the fatty substance called tyloses on the surface of fiber. Further, these holes were hidden inside the surfaces of the untreated fiber. Prasad et al. 1983 also reported similar results. Individual cellular elements can be seen on the surface of clean fine fiber on alkali treatment. It was also found that roughness of the surface increased with increase in concentration of aqueous sodium hydroxide. The rough surface morphology is typical for the treated fibers, because of the removal of hemicelluloses and other surface impurities. Thus, alkali treatment resulted in significant change of morphology of the fiber surface leading to increased wettability and for composites; this normally results in better bonding through a kind of mechanical interlocking between the matrix and the roughened fiber surface.

The composition of the fibers was determined by following chemical analysis procedure reported in recent literature (Moran et al. 2008). Table 1 shows the chemical composition of untreated and alkali treated fibers. An analysis of the results of Table 1 revealed that the alkali treatment changed the fiber composition concerning the three main components (α-cellulose, hemicellulose and lignin). The hemicellulose composition was effected by the concentration of the alkaline solution, as hemicellulose ais much more sensitive to the action of aqueous sodium hydroxide at room temperature than lignin or α -cellulose (Gassan and Bledzki 1997). The diameter of the fine fibers after alkali treatment and corresponding aspect ratio (L/D) values are presented in Table 2. The decrease in diameter might be due to the removal of the hemicellulose present on the surface of the fibers by alkali. This was in support of the chemical analysis data of the alkali treated fibers as shown in Table 1. The hemicellulose content of the borassus fine fibers was reduced from 29.6% to 9.66% for different concentrated alkali solution with treatment and because of this, % of α-Cellulose and lignin content increased. Further, the decrease in diameter was proportional to the concentration of the alkali solution. The chemical composition of Borassus fibres was comparable with that of sisal, jute, tamarind, sponge gourd and banana.

Table 1. Chemical composition of untreated and alkali treated Borassus fine fibers

Alkali con-	Component (%)					
centration	α-Cellulose [SD]	Hemicellulose [SD]	Lignin [SD]			
Untreated	53.40 [1.2]	29.60 [1.4]	17.00 [1.5]			
2%	56.99 [0.9]	23.67 [1.2]	19.33 [1.1]			
5%	60.02 [1.4]	17.00 [0.7]	22.00 [0.5]			
10%	62.32 [1.0]	13.31 [0.5]	24.66 [0.8]			
15%	63.66 [0.4]	10.66 [1.3]	25.66 [1.8]			
20%	62.33 [0.2]	9.66 [1.1]	28.00 [1.6]			

SD: Standard Deviation



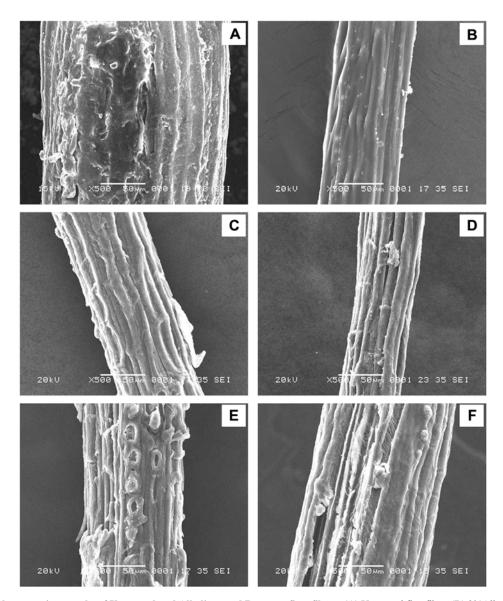


Fig. 1 Scanning electron micrographs of Untreated and Alkali treated Borassus fine fibers: (A) Untreated fine fiber; (B) 2%Alkali treated fine fiber; (C) 5%Alkali treated fine fiber; (E) 15%Alkali treated fine fiber; (F) 20% Alkali treated fine fiber

Table 2. Physical properties of untreated and alkali treated Borassus fine fibers

Fibers treated with	Average	Average Diameter	Aspect	Crystallin-
аткап	Length L (cm)	Diameter D (cm)	ratio (L/D)	ity Index (%)
Untreated	10	0.01408	710	38.3
2% Alkali treated	10	0.01355	738	39.0
5% Alkali treated	10	0.01232	811	47.4
10% Alkali treated	10	0.01124	889	58.2
15% Alkali treated	10	0.00996	1004	67.4
20% Alkali treated	10	0.00904	1106	41.5

FT-IR spectroscopy is an appropriate technique to establish the variations introduced by the different alkali treatments on the chemical structure of borassus fine fibers. FT-IR spectra of the untreated and alkali treated fine fibers are presented in Fig. 2. Both untreated and alkali treated fibers presented the typical vibration bands of the components mainly corresponding to cellulose, hemicellulose and lignin. All vibrations are summarized in Table 3. Spectra of the untreated fibers showed the presence of a strong absorption band for intermolecularly bonded hydroxyl (O-H) groups at around 3443 cm⁻¹. The appearance of very intense and broad absorption was a clear indication of many hydroxyl groups in the fiber (Saha et al. 1991; Pandey and Pitman. 2003). This observation supported that the hydroxyl groups were involved in the hydrogen bonding. The C-H stretching vibrations of methylene/ methylene units of all three constituents were seen as two shoulder peaks at 2925 and 2858 cm⁻¹ (Sun et al. 2005). In the 1750-900 cm⁻¹ region, many absorption peaks were noticed whose intensities varied from low, moderate to high. Among them, notable were 1744, 1650, 1637, 1458, 1426, 1379, 1339,



1317, 1252, 1161, 1056 and 891cm⁻¹ values (Table 3). A broad, medium intensity ester carbonyl vibration appeared at 1744 cm⁻¹, which was presumed to carbonyl (C=O) stretching of acetyl groups in hemicellulose (Pandey 1999). The close examination of absorption at 1744 cm⁻¹ indicated the presence of a shoulder signal at 1718 cm⁻¹, which was arising from acid carbonyl. Another vibration at 1650 cm⁻¹ arose when the semi-acetal group of the cellulose molecule transformed into aldehyde and with a shoulder at 1637 cm⁻¹. This was attributed to carbonyls of ketonic groups typically present in the lignin component. The aromatic ring C=C stretching vibrations were noted in the region 1605-1510 and the intensities of them was very low. The medium intensity bands around 1458 cm⁻¹, 1426 cm⁻¹, 1379 and 1339 cm⁻¹ were associated with methylene deformation, methyl asymmetric, and symmetrical vibrations and O-H in plane deformation respectively (Ray and Sarkar. 2001; Mwaikambo and Ansell. 2002). The characteristic C-O-C stretching vibration of ester groups of hemicellulose was seen at 1259 cm⁻¹ (Pandey. 1999) whereas asymmetrical stretching of C-O-C in the cellulose and hemicellulose at 1161cm⁻¹ and the C-O stretching of hydroxyl functionality was observed at 1056 cm⁻¹ (Pandey. 1999; Liu et al. 2009). Finally, the vibrations appearing at 891cm⁻¹ were from di-substituted ring stretching and out-of-plane carbon

single bonded hydrogen (Mwaikambo and Ansell. 2002).

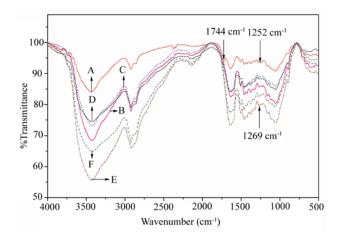


Fig. 2 FT-IR spectra of Untreated and Alkali treated Borassus fine fibers. (A) Untreated fine fibers; (B) 2%Alkali treated fine fibers; (C) 5%Alkali treated fine fibers; (D) 10%Alkali treated fine fibers; (E) 15%Alkali treated fine fibers; (F) 20% Alkali treated fine fibers

Table 3: Absorption bands for functional groups of Cellulose, Hemicellulose and Lignin in untreated and alkali treated Borassus fine fibers

Absorbance in (cm ⁻¹)			_			
Untreated	2% Alkali treated	5%Alkali treated	10% Alkali treated	15%Alkali treated	20% Alkali treated	Possible Assignment
3443	3436	3436	3448	3441	3441	O-H Stretching(cellulose & hemicellulose)
2925	2923	2923	2923	2923	2923	C-H Stretching (cellulose & hemicellulose)
2858	2857	2857	2857	2857	2852	CH ₂ Symmetric Stretching (cellulose & hemicellulose)
1744						C=O Stretching of (carbonyl ester)
1718						C=O Stretching of (carboxylic acid)
1650	1648	1650	1650	1650	1647	Aldehyde (CHO) (lignin)
1637	1640	1638	1639	1635	1639	Ketonic (-C=O) (lignin)
1509	1508	1500	1508	1508	1508	Aromatic skeletal vibration (lignin)
1458	1461	1463	1460	1463	1462	C-H deformation (methyl methylene)
1426	1425	1438	1425	1427	1427	CH ₂ asymmetric stretching (lignin)
1379	1375	1382	1378	1376	1379	C-H symmetrical deformation cellulose
1339	1339	1325	1340	1332	1338	O-H in-plane bending
1252	1265	1264	1265	1264	1269	C-O Stretching of acetyl (hemicellulose)
1161	1162	1164	1160	1160	1161	C-O-C Asymmetrical stretching (cellulose &hemicellulose)
1056	1054	1051	1055	1054	1063	C-O/ C-C Stretching vibration
893	893	891	893	892	893	β-Glucosidic linkage

The examination of the spectra of different concentrated alkali treated fine fibers showed peaks at 3436, 2923 and 2857cm⁻¹, which were assigned to O-H, C-H (asym and sym) stretching vibrations, respectively. However, the characteristic 1744 and 1718 cm⁻¹ primarily due to CH₃COO and COOH functionalities of hemicellulose noted for the raw fiber was now absent. The disappearance of these characteristic stretching vibrations clearly indicated that the alkali treatment had significantly removed the hemicellulose content. This was further supported by the chemical analysis of alkali treated fibers. On the other hand, the appearance of 1648 and 1640 cm⁻¹, indicated that the lignin component was intact. The disappearance of ester carbonyl stretching

vibrations was further supported by reduction in the intensity of 1259 cm⁻¹ (C-O-C), but on treatment with higher concentrations (15 and 20%) of alkali, this band was shifted to 1265cm⁻¹ (1259 cm⁻¹ in untreated fiber), which was a clear indication of change in cellulosic structure (Reddy et al. 1990). For other vibrations, i.e., 1461, 1425, 1375, 1342, 1162, 1053, 893 cm⁻¹ no appreciable changes were noticed. Thus, the FTIR studies suggested the reduction of the hemicellulose content on alkali treatment of the fibers. This was in support of the chemical analysis data of the alkali treated fibers as shown in Table 1.

The wide angle X-Ray diffraction patterns of untreated and different alkali concentrated treated fibers are shown in Fig. 3.



The diffractograms showed two reflections, corresponding to 2θ values of around 16° and 22° respectively. Among these, the low angle reflection (16°) was of low intensity, representing $I_{(am)}$ of amorphous material and the other reflection (22°) had higher intensity, and it represented $I_{(002)}$ of crystalline material in cellulosic fibers. Similar observations were also made for other natural fibers (Ray et al. 2001, Mwaikambo et al. 2002 and Wang et al. 2003). The degree of crystallinity(I_C) was calculated (Mwaikambo et al. 2002) using Eq. (1).

$$I_C = \frac{[I_{(002)} - I_{(am)}]}{I_{(002)}} \times 100$$
 (2)

The extent of crystallinity formation by sodium hydroxide

treatment was also determined from the improvement in the intensity of the peaks. Table 2 showed that the degree of crystallinity increased after alkali treatment. This might be due to the resulting close packing of cellulose chains due to the removal of hemicelluloses, which normally separated the cellulose chains. We observed that when the fibers were treated with alkali of concentrations 2% to 15%, the crystalline cellulose content increased. However, when treated with 20% of alkali, the amount of the crystalline cellulose rapidly dropped indicating the molecular degradation of cellulose at this concentration. Das and Chakraborty (Das and Chakraborty 2008) made similar observation in the case of bamboo fibers.

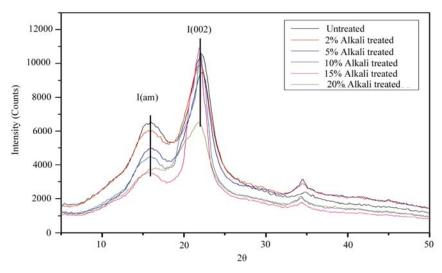


Fig. 3 X-Ray diffractograms of untreated and alkali treated Borassus fine fibers

Table 4: Tensile properties of untreated and alkali treated Borassus fine fibers

Alkali treatment	Young's Modulus (GPa) [SD]	IM (%)	Strength (MPa) [SD]	IM (%)	Elongation at break (%) [SD]	IM (%)
Untreated	10.8 [0.4]		70.8 [4.1]		34.8 [3.5]	
2% Alkali treated	12.5 [0.2]	13.5	78.4 [1.1]	9.7	42.4 [1.1]	17.9
5% Alkali treated	18.6 [0.1]	42	92.1 [0.7]	23.1	41.2 [0.4]	15.5
10% Alkali treated	21.6 [0.4]	49.9	95.1 [2.8]	25.5	53.2 [1.2]	34.5
15% Alkali treated	25.7 [0.6]	58	100.1 [5.4]	29.2	41.6 [1.0]	16.3
20% Alkali treated	20.1 [0.1]	46.2	57.1 [6.4]	-23.9	45.2 [3.2]	23

SD: Standard Deviation, IM: Increment

The tensile properties of untreated and alkali treated borassus fine fibers are presented in Table 4. From the table it can be seen that the tensile modulus, tensile strength and %elongation increased with increasing alkali concentration. Change in mechanical properties were possibly due to interacting factors such as the rupture of alkali-sensitive bonds existing between the different components of the fiber as a result of removal of the hemicelluloses by which the fiber became more homogeneous, for instance the stress transfer between ultimate cells improved (Mukherjee et al. 1993). Further, in the case of untreated fibers, hemicellulose remained dispersed in the interfibrillar region separating cellulose chains from one another and because of this barrier, these

chains were in a state of strain. When this barrier hemicellulose was removed, formation of new hydrogen bonds between cellulose chain resulted and the fibrils became more capable of rearranging themselves in a more compact manner resulting in a close packing of the fibers (Wang et al. 2003). The increase in fiber strength for treatments with 2% to 15% alkali solutions could be attributed to an increase in packing density and molecular orientation due to the removal of cementing materials (hemicellulose and waxes). The decrease in strength for treatment with 20% solution was attributed to the degradation of cellulose at higher concentrations of alkali.

The primary thermograms of untreated and different concen-



trated alkali treated fibers are presented in Fig. 4 and it can be observed that the thermal degradation of borassus fine fibers was a three - step process. The first, second and third stages were found in the temperature ranges of 50-125°C, 220-300°C and 300 – 400 °C respectively. Due to the differences in the chemical structures, the components of wood fibers usually decomposed at different temperatures (Yang et al. 2007). The first decomposition temperature range of 50-25°C corresponded to the evaporation of moisture. The alkali treated fibers presented a slightly lower in moisture content than that of the untreated fibers, which might be due to reduction of free hydroxyl groups. The second decomposition step of untreated fibers in the temperature range of 220 – 300°C indicated the loss of hemicellulose and some part of the lignin. The third decomposition step corresponding to the decomposition of cellulose was observed in the range of 300 -400°C. Similar observation was also made in the case of many natural fibers (Reddy et al. 2009a; Rajulu et al. 2002). From Fig. 4, it can be observed that the thermograms of the fibers showed a similar trend under all conditions. Using these thermograms, the degradation temperatures and char content were noted down as

per Doyle. 1985 method. According to him, the initial and final degradation temperatures are the temperatures corresponding to the intersection of the tangents drawn at the initial and final curvature portions of the thermograms. The decomposition temperatures of untreated and mercerized treated fibers showed remarkable differences. These values are presented in Table 5. From this table, it is evident that the thermal stability of the alkali treated fibers was found to be higher than that of untreated fibers. The residual char content had increased from 16.5% to 24.1% on alkali treatment. A similar observation was reported by Reddy et al. 2009a who explained that alkalization reduced the hemicellulose to a considerable extent, giving rise to lignin-cellulose complex, thereby making the product more stable than the untreated sample, and this was reflected in the increased amount of residual char. The thermal stability of the fibers treated with 20% alkali solution was found to be lower even to that of untreated fibers. This is in support of the earlier observation in other properties of the fibers treated with 20% alkali solution indicating the degradation of cellulose at this concentration.

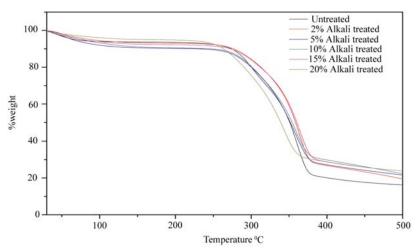


Fig. 4 Primary thermograms of untreated and alkali treated Borassus fine fiber

Table 5: Thermal degradation parameters of untreated and alkali treated Borassus fine fibers

Fibers treated with	Initial degradation	Final degradation	Char content	
alkali	Temperature (^O C)	Temperature (^O C)	(%)	
Untreated	294	371	16.5	
2% Alkali treated	296	372	19.4	
5% Alkali treated	301	372	21.6	
10% Alkali treated	308	369	22.4	
15% Alkali treated	305	372	24.1	
20% Alkali treated	281	350	24.1	

Conclusions

The Borassus fruit fine fibers were extracted from the fruit by water treatment. The composition of the untreated and alkali treated fibers with different concentrations was determined by chemical analysis, which indicated the presence of α-cellulose, hemicellulose, and lignin. The alkali treatment of fibers resulted in solubilization of hemicellulose leading to reduction of its content in the fibers. FT-IR provided additional information on the reactivity of fibers following treatment by alkali. This technique also enabled to identify and confirm the effective removal of chemical groups (hemicellulose) upon alkalization. The alkali treatment of untreated fine fibers caused the transformation of lower crystalline cellulose into higher crystalline cellulose. The effectiveness of transformation depended on the concentration of sodium hydroxide solution. The alkali treated fibers showed an increase in crystallinity index, for sodium hydroxide concentrations up to 15% and this index decreased at higher concentration (20%). The tensile properties of the fibers improved when the fibers were treated with alkali solution of up to 15% concentration and decreased thereafter. The thermal stability of the fibers improved slightly by alkali treatment. The residual char formation was enhanced in the treated fibers. It was established that



15% alkali treated borassus fine fibers were successful candidates for making polymer composites.

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